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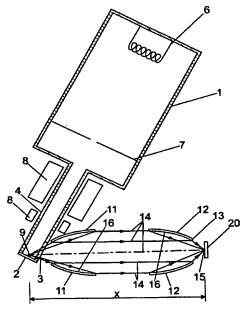
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(54) Title: X-RAY FLUORESCENCE APPARATUS



(57) Abstract: This invention relates to a portable apparatus for carrying out X-ray fluorescence spectrometry on specimen materials at a distance from the apparatus. The apparatus comprises an X-ray generating tube (1), such as a microfocus tube, and two paraboloidal X-ray reflecting mirrors. The first collecting mirror (11) is positioned in close coupled arrangement adjacent to the exit window (3) of the tube (1), such that it emits parallel X-ray radiation (14) to the second focusing mirror (12) which is aligned on the axis of and spaced apart from the first mirror (11). The second mirror (12) collects the parallel X-ray radiation (14) at its end closest to the first mirror (11) and emits X-ray radiation in a focused beam onto the specimen (20). The distance between the first and second mirrors is adjusted to suit the distance from the X-ray tube (1) to the specimen (20). Focal spots on the specimen (20) of diameter less than 15 µm are possible, enabling precise analysis of small areas of the specimen.



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carrying out X-ray fluorescence spectrometry (XRF), 4 and particularly to a portable apparatus which is 5 able to generate X-ray fluorescence in materials at a 6 7 distance from the apparatus. 8 X-ray fluorescence spectrometry is a non-destructive 9 technique for determining the elemental composition 10 of a wide variety of materials. X-ray fluorescence 11 (XRF) is the secondary emission of X-rays at 12 wavelengths characteristic of each element present 13 when a material is irradiated with a primary X-ray 14 beam. In commercially available XRF spectrometers 15 the bulk sample is usually irradiated directly by X-16 rays from a sealed tube. The technique is 17 sufficiently sensitive to detect elements which are 18 present at concentrations as low as one or two parts

per million. There is however a requirement for

This invention relates to an apparatus and method for

2

1 greater sensitivity in applications in which it is

- 2 desired to examine small areas on bulk samples or
- 3 where the sample itself is small. The type of
- 4 instrumentation required for this technique is
- 5 sometimes called Micro X-ray Fluorescence Analysis
- 6 (MXRFA or MXA) apparatus.

7

- 8 Several methods presently exist for MXRFA. Among
- 9 them is the use of mono-capillary and poly-capillary
- 10 X-ray focusing optics coupled to standard or
- 11 microfocus X-ray generating tubes. These suffer from
- the drawback that samples have to be placed very
- 13 close to the output of the optic (generally less than
- 14 300 μ m). The minimum focal spot generally
- 15 commercially available with polycapillaries is 28 μm.
- 16 This is relatively large and limits the fineness of
- 17 the resolution with which areas of a sample can be
- 18 analysed.

19

- 20 Another method which presently exists for MXRFA is to
- use a synchrotron in conjunction with Fresnel lenses.
- 22 Such apparatus is massive and not portable, although
- 23 beams having a focal spot of only 1 μm can be
- 24 achieved, giving greater accuracy in analysis of
- 25 samples. This method suffers from the disadvantage
- 26 that synchrotron radiation sources are large fixed
- facilities which are not portable and are not
- available in most laboratories, so cannot be accessed
- on a routine basis.

3

1 A further method of MXRFA which exists is the use of

- 2 a synchrotron in conjunction with mono-capillary
- 3 lenses. Such apparatus is also not portable, and
- 4 beam sizes are limited to a focal spot of 5-10 μm .

5

- 6 It is therefore an object of the invention to provide
- 7 an apparatus for carrying out X-ray fluorescence
- 8 spectrometry which is portable yet which is capable
- 9 of analysing samples of are less than 30 $\mu m\,.$

10

- 11 According to a first aspect of the present invention
- there is provided an apparatus for carrying out X-ray
- 13 fluorescence spectrometry comprising an X-ray
- 14 generating tube and two paraboloidal X-ray reflecting
- mirrors, the generating tube having an X-ray source
- 16 and an X-ray exit window through which X-ray
- 17 radiation from said source is emitted,
- the first mirror being aligned on a first axis and
- 19 positioned in close coupled arrangement adjacent to
- the exit window, the second mirror being aligned on
- 21 said first axis and being positioned in spaced apart
- 22 relationship to the first mirror,
- 23 the first mirror being adapted to collect diverging
- 24 X-ray radiation at its first end adjacent to the
- 25 collecting window and to emit X-ray radiation in a
- 26 substantially parallel beam at its second end,
- 27 the second mirror being adapted to collect
- 28 substantially parallel X-ray radiation at its first
- 29 end closest to the first mirror and to emit X-ray
- radiation in a focused beam at its second end.

4

1 By using first and second mirrors in this way, the

- 2 focal spot on the target of the X-ray tube is
- 3 transferred to the image plane, at unity
- 4 magnification. The focal spot at the image plane on
- 5 the sample subjected to fluorescence has a high
- 6 brightness, and focal spots on the sample of diameter
- 7 less than 15 μm are possible.

8

- 9 Preferably the first and second mirrors are
- 10 cylindrical specularly reflecting mirrors.
- 11 Preferably the first end of the first mirror is
- positioned between 5 and 50 mm from the X-ray source.

13

- 14 Preferably the apparatus further comprises a housing
- 15 containing the first and second mirrors.

16

- 17 The second mirror may be fixed in position relative
- 18 to the first mirror.

19

- 20 Alternatively the second mirror may be movable in
- 21 position relative to the first mirror. The apparatus
- 22 may further comprise a guide means for guiding said
- 23 second mirror in a direction parallel to the first
- 24 axis, and adjustment means for adjusting the spacing
- of the first and second mirrors.

26

- 27 The apparatus may further comprise angular adjustment
- 28 means adapted to allow angular adjustment of the
- 29 mirror housing with the X-ray generator tube.

5

Preferably the X-ray generator tube is adapted to

- produce an X-ray source at the target having a
- 3 maximum width of less than 50 μ m, more preferably
- 4 less than 15 μ m.

5

- 6 According to a second aspect of the present invention
- 7 there is provided a method of delivering X-ray
- 8 radiation to a specimen for the purpose of X-ray
- 9 fluorescence spectrometry using an X-ray generating
- 10 tube, the generating tube having an X-ray exit window
- through which X-ray radiation is emitted,
- the method comprising placing first and second
- 13 paraboloidal X-ray reflecting mirrors between the
- 14 exit window and the specimen,
- using the first mirror to collect diverging X-ray
- 16 radiation at its first end adjacent to the exit
- 17 window and to emit X-ray radiation in a substantially
- 18 parallel beam at its second end,
- 19 and using the second mirror to collect substantially
- 20 parallel X-ray radiation at its first end closest to
- 21 the first mirror and to emit X-ray radiation at its
- 22 second end to a focused spot on the specimen.

23

- 24 Preferably the method uses an apparatus according to
- 25 the first aspect of the invention.

26

- 27 Embodiments of the invention will now be described,
- 28 by way of example only, with reference to the
- 29 accompanying figures, where:

Fig. 1 is a schematic view of two X-ray focusing 1 mirrors used in accordance with the invention to 2 focus an X-ray beam from the source on the X-ray 3 target to the sample to be subject to X-ray 4 5 fluorescence spectrometry; 6 Fig. 2 is a schematic view of an apparatus according 7 to a first aspect of the invention having mirrors 8 fixed relative to each other; and 9 10 Fig. 3 is a schematic view of an apparatus according 11 to a second aspect of the invention having mirrors 12 adjustable relative to each other. 13 14 Referring to Fig. 1 there is shown, in a schematic 15 form and not to scale, an X-ray generating tube 1 16 having an exit window 3, an electron source 6, an 17 anode 7, focusing and stigmator coils 8 and a target 18 2 on which is formed an X-ray source 9. A suitable 19 X-ray generating tube is the MICROSOURCE™ tube 20 described in International Patent Application No 21 PCT/GB97/02022, which is a compact X-ray generator 22 capable of producing small-size, high intensity X-ray 23 sources for low power input. Typically the exit 24 window 3 of the generator 1 is provided in the narrow 25 portion 4 of the X-ray tube about which the X-ray 26 focusing coils 8 are arranged, to the side of the X-27 ray target 2. A first X-ray focusing mirror 11, the 28 collection mirror, is positioned adjacent to the exit 29 window 3 in close coupled arrangement, and a second 30

X-ray focusing mirror 12, the focusing mirror, is

7 arranged coaxially with the first X-ray focusing 1 mirror 11, to transfer the . Suitable mirrors 11, 12 2 are MICROMIRROR™ X-ray optics as supplied by Bede 3 Scientific Instruments Ltd. The mirrors are 4 cylindrical specularly reflecting mirrors. Each 5 mirror comprises a cylindrical body having an axially 6 symmetrical passage extending therethrough. There is 7 an aperture at each end of the body which 8 communicates with the passage. The reflecting 9 surface is on the inside of the long axis of the 10 cylinder and has a shape corresponding to a 11 paraboloid of revolution about the long axis of the 12 cylinder. 13 14 A paraboloidal profile produces an almost parallel, 15 essentially non-divergent beam 14. The interior 16 reflecting surface 16 is coated in an exceptionally 17 smooth coating of gold or similar in order to provide 18 specular reflectivity. Typically the mirror is made 19 of nickel and is of the order of 10 to 100 mm in 20 length, typically about 30 mm. The outside diameter 21 of the mirror is typically 6 mm. The internal 22 diameter is typically less than 4 mm. The entry 23 aperture is generally smaller than the exit aperture. 24 25 The two mirrors have an identical profile. 26

source to first mirror distance is in the range 5 to

27 28

29

50 mm.

Typically the X-ray generator produces a sub-15 μm 1 spot source on a target of less than 10 mm diameter 2 at a power of up to 30 W. 3 The first mirror or paraboloidal optic 11 has a high 5 angle of collection and reflects X-rays into a 6 substantially parallel beam. In practice a beam of 7 divergence less than 40 arc seconds can be achieved. 8 9 The second mirror or paraboloidal optic 12 takes the 10 parallel beam and focuses it down to a spot 15 on the 11 specimen 20 of a size similar to that of the X-ray 12 source, typically a spot with a diameter of less than 13 15 µm. 14 15 The focus 15 of the second optic 12 is typically 16 about 10 to 20 mm away from the far end 13 of optic, 17 giving a much more convenient working distance than 18 is available from prior art XRF apparatus, such as 19 monocapillaries. 20 21 The distance between the two optics 11, 12 may be 22 continuously changed without affecting the focal spot 23 quality, thereby allowing a range of source to sample 24 distances X to be achieved. Typically distance X 25 will be 100 mm or more. 26 27 X-ray optics have very well defined profiles and low 28 surface roughness, and therefore work at very high 29

efficiency. By using paraboloidal mirrors the

apparatus of the invention achieves broad band

30

transmission of X-rays, with an efficiency close to

- 2 1, since only double reflection of the X-ray
- 3 radiation is required.

4

- 5 The invention achieves high X-ray brightness at the
- 6 focal plane on the target, with a focal spot diameter
- 7 of as low as 10 μ m.

8

- 9 The apparatus of the invention is truly portable,
- 10 giving it applications in areas such as forgery
- 11 detection, which require the apparatus to be taken to
- 12 the specimen.

13

- 14 A parabolic surface will produce a parallel beam if
- 15 the source is placed at the focal point. Conversely
- 16 a focused beam will be brought to a focus when a
- 17 parabolic surface is illuminated with a parallel
- 18 beam. Therefore the method and apparatus of the
- invention serves to transfer the image of the X-ray
- 20 spot from the target to the specimen. It should be
- 21 noted that the target may not be perpendicular to the
- 22 axis of the of the mirrors, so that the effective
- dimension of the image on the target, when viewed
- 24 along the axis of the mirrors, is less than the
- 25 actual dimension on the target.

- 27 The focal spot size at the specimen is thus primarily
- 28 determined by the spot size on the target of the X-
- 29 ray tube. Since the first mirror produces a parallel
- 30 beam, the focal spot size at the specimen is, within
- 31 practical limits, independent of the distance of the

10

second mirror along the beam axis. Therefore the
second mirror can be placed at the required distance
from the first in order to suit the geometrical
requirements of the equipment.

5

Figs. 2 and 3 show two schematic arrangements for housing the apparatus of the invention.

8

In the simplest case, shown in Fig. 2, the collector 9 and focusing mirrors 11, 12 are aligned with each 10 other and are fixed within a cylindrical housing 30. 11 The housing is aligned relative to the X-ray source 12 9, shown purely schematically in Figs. 2 and 3, on 13 the beam axis 32, either fixedly or adjustably. 14 housing 30 may be subject to a partial or total 15 vacuum, to improve the efficiency of the mirrors and 16 reduce energy absorption as the X-rays pass through 17 the gas in the housing 30. It is to be understood 18 that in practice the source 9 is part of an X-ray 19

generating tube 1 (not shown in Figs. 2 and 3).

20 21

In use the housing 30 is placed adjacent to the X-ray 22 source, and a control mechanism 35 allows fine 23 adjustment of the position of the housing 30 in the 24 x, y and z directions so that the axis 32 of the 25 mirrors is accurately aligned with the X-ray source 9 26 and directed to the specimen 20. 27 mechanism 35 may comprise any suitable mechanisms 28 which permit fine translational adjustment, such as 29 lead screws or Vernier controls. 30

In the example of Fig. 3, each mirror 11, 12 is 1 provided with a separate housing 40, 41. 2 housings 40, 41 may further be contained in an outer 3 housing, not shown, which may be partially or 4 completely evacuated. The apparatus allows alignment 5 of the second mirror 12 relative to the first mirror 6 11 and translation of the second mirror 12 along the 7 beam axis 43 by means of control mechanism 44. 8 9 Alignment of the whole mirror assembly relative to 10 the X-ray source 9 is possible by means of control 11 mechanism 45. Mechanisms 44 and 45 are similar to 12 mechanism 35 described with reference to Fig. 2, and 13 are not described further. 14 15 Although the invention has been described with 16 reference to a microfocus X-ray generator, the 17 invention can be used with any suitable X-ray 18 generator which is capable of producing a small 19 source of sufficient intensity. 20 21 The mirror housing 30, 40 may be attached to the X-22 ray tube 1 or may be positioned independently. 23 24 These and other modifications and improvements can be 25

incorporated without departing from the scope of the

26

27

invention.

1 CLAIMS

2

- 3 1. An apparatus for carrying out X-ray fluorescence
- 4 spectrometry comprising an X-ray generating tube (1) and
- 5 two paraboloidal X-ray reflecting mirrors (11, 12), the
- 6 generating tube having an X-ray source (9) and an X-ray
- 7 exit window (3) through which X-ray radiation from said
- 8 source is emitted,
- 9 the first mirror (11) being aligned on a first axis (32,
- 10 43) and positioned in close coupled arrangement adjacent
- 11 to the exit window (3), the second mirror (12) being
- 12 aligned on said first axis and being positioned in spaced
- apart relationship to the first mirror (11),
- 14 the first mirror (11) being adapted to collect diverging
- 15 X-ray radiation at its first end adjacent to the
- 16 collecting window (3) and to emit X-ray radiation in a
- 17 substantially parallel beam at its second end,
- 18 the second mirror (12) being adapted to collect
- 19 substantially parallel X-ray radiation at its first end
- 20 closest to the first mirror and to emit X-ray radiation
- in a focused beam at its second end.

22

- 23 2. An apparatus according to Claim 1, wherein the first
- 24 and second mirrors (11, 12) are cylindrical specularly
- 25 reflecting mirrors.

26

- 27 3. An apparatus according to Claim 1 or 2, wherein the
- 28 first end of the first mirror (11) is positioned between
- 5 and 50 mm from the X-ray source (9).

- 31 4. An apparatus according to any preceding Claim,
- 32 wherein the apparatus further comprises a housing
- 33 containing the first and second mirrors.

2 5. An apparatus according to any preceding Claim,

3 wherein the second mirror is fixed in position relative

4 to the first mirror.

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6 6. An apparatus according to any of Claims 1 to 4,

7 wherein the second mirror is movable in position relative

8 to the first mirror.

9

10 7. An apparatus according to Claim 6, further

11 comprising a guide means for guiding said second mirror

in a direction parallel to the first axis, and adjustment

13 means for adjusting the spacing of the first and second

14 mirrors.

15

16 8. An apparatus according to any preceding Claim,

17 further comprising angular adjustment means adapted to

18 allow angular adjustment of the mirror housing with the

19 X-ray generator tube.

20

21 9. An apparatus according to any preceding Claim,

22 wherein the X-ray generator tube is adapted to produce an

23 X-ray source at the target having a maximum width of less

24 than 50 μ m, more preferably less than 15 μ m.

25

26 10. An apparatus according to any preceding Claim,

27 wherein the apparatus is portable and the X-ray generator

28 is a microfocus generator.

29

30 11. A method of delivering X-ray radiation to a specimen

31 for the purpose of X-ray fluorescence spectrometry using

an X-ray generating tube, the generating tube having an

1 X-ray exit window through which X-ray radiation is

- 2 emitted,
- 3 the method comprising placing first and second
- 4 paraboloidal X-ray reflecting mirrors between the exit
- 5 window and the specimen,
- 6 using the first mirror to collect diverging X-ray
- 7 radiation at its first end adjacent to the exit window
- 8 and to emit X-ray radiation in a substantially parallel
- 9 beam at its second end,
- 10 and using the second mirror to collect substantially
- 11 parallel X-ray radiation at its first end closest to the
- 12 first mirror and to emit X-ray radiation at its second
- end to a focused spot on the specimen.

14

- 15 12. A method according to Claim 11, wherein the first
- and second mirrors (11, 12) are cylindrical specularly
- 17 reflecting mirrors.

18

- 19 13. A method according to Claim 11 or 12, wherein the
- 20 first end of the first mirror (11) is positioned between
- 21 5 and 50 mm from the X-ray source (9).

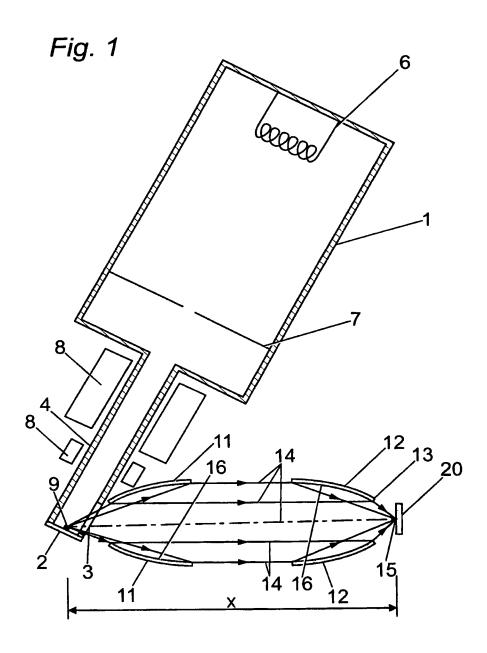
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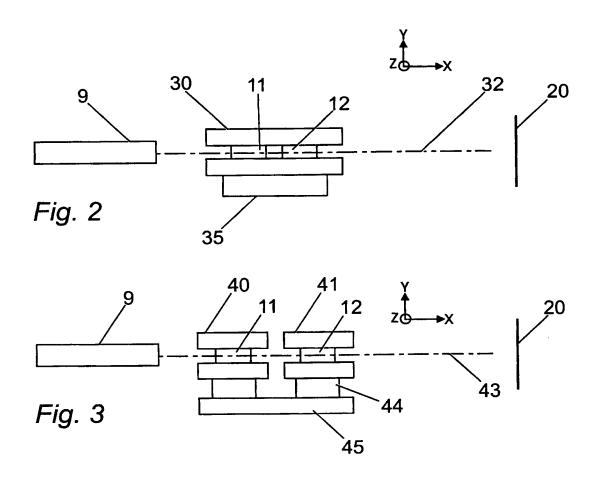
- 23 14. A method according to any one of Claims 11 to 13,
- 24 wherein the focused spot on the specimen has a maximum
- 25 dimension of 50 μ m.

26

- 27 15. A method according to any one of Claims 11 to 14,
- 28 further comprising the step of adjusting the spacing of
- 29 the first and second mirrors to produce a focused spot on
- 30 the specimen.

- 32 16. A method according to any one of Claims 11 to 15,
- 33 wherein the X-ray generator is a microfocus generator.





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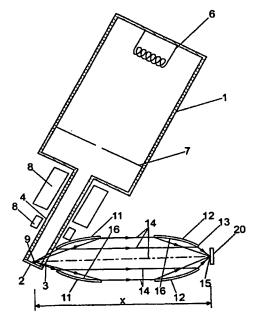
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(54) Title: X-RAY FLUORESCENCE APPARATUS



(57) Abstract: This invention relates to a portable apparatus for carrying out X-ray fluorescence spectrometry on specimen materials at a distance from the apparatus. The apparatus comprises an X-ray generating tube (1), such as a microfocus tube, and two paraboloidal X-ray reflecting mirrors. The first collecting mirror (11) is positioned in close coupled arrangement adjacent to the exit window (3) of the tube (1), such that it emits parallel X-ray radiation (14) to the second focusing mirror (12) which is aligned on the axis of and spaced apart from the first mirror (11). The second mirror (12) collects the parallel X-ray radiation (14) at its end closest to the first mirror (11) and emits X-ray radiation in a focused beam onto the specimen (20). The distance between the first and second mirrors is adjusted to suit the distance from the X-ray tube (1) to the specimen (20). Focal spots on the specimen (20) of diameter less than 15 μm are possible, enabling precise analysis of small areas of the specimen.



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X-ray Fluorescence Apparatus

1

This invention relates to an apparatus and method for 3 carrying out X-ray fluorescence spectrometry (XRF), 4 and particularly to a portable apparatus which is 5 able to generate X-ray fluorescence in materials at a 6 distance from the apparatus. 7 8 X-ray fluorescence spectrometry is a non-destructive 9 technique for determining the elemental composition 10 of a wide variety of materials. X-ray fluorescence 11 (XRF) is the secondary emission of X-rays at 12 wavelengths characteristic of each element present 13 when a material is irradiated with a primary X-ray 14 In commercially available XRF spectrometers 15 the bulk sample is usually irradiated directly by X-16 rays from a sealed tube. The technique is 17 sufficiently sensitive to detect elements which are 18 present at concentrations as low as one or two parts 19 per million. There is however a requirement for 20

2 greater sensitivity in applications in which it is 1 desired to examine small areas on bulk samples or 2 where the sample itself is small. The type of 3 instrumentation required for this technique is 4 sometimes called Micro X-ray Fluorescence Analysis 5 6 (MXRFA or MXA) apparatus. 7 Several methods presently exist for MXRFA. Among 8 them is the use of mono-capillary and poly-capillary 9 X-ray focusing optics coupled to standard or 10 microfocus X-ray generating tubes. These suffer from 11 the drawback that samples have to be placed very 12 close to the output of the optic (generally less than 13 The minimum focal spot generally 14 $300 \mu m)$. commercially available with polycapillaries is 28 $\mu\text{m}\,.$ 15 This is relatively large and limits the fineness of 16 the resolution with which areas of a sample can be 17 analysed. 18 19 Another method which presently exists for MXRFA is to 20 use a synchrotron in conjunction with Fresnel lenses. 21 Such apparatus is massive and not portable, although 22 beams having a focal spot of only 1 μm can be 23 achieved, giving greater accuracy in analysis of 24 This method suffers from the disadvantage 25 samples. that synchrotron radiation sources are large fixed 26

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1 A further method of MXRFA which exists is the use of

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- 3 lenses. Such apparatus is also not portable, and
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- 16 and an X-ray exit window through which X-ray
- 17 radiation from said source is emitted,
- 18 the first mirror being aligned on a first axis and
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- 20 the exit window, the second mirror being aligned on
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- 26 substantially parallel beam at its second end,
- 27 the second mirror being adapted to collect
- 28 substantially parallel X-ray radiation at its first
- 29 end closest to the first mirror and to emit X-ray
- radiation in a focused beam at its second end.

By using first and second mirrors in this way, the 1 focal spot on the target of the X-ray tube is 2 transferred to the image plane, at unity 3 magnification. The focal spot at the image plane on 4 the sample subjected to fluorescence has a high 5 brightness, and focal spots on the sample of diameter 6 less than 15 µm are possible. 7 8 Preferably the first and second mirrors are 9 cylindrical specularly reflecting mirrors. 10 Preferably the first end of the first mirror is 11 positioned between 5 and 50 mm from the X-ray source. 12 13 Preferably the apparatus further comprises a housing 14 containing the first and second mirrors. 15 16 The second mirror may be fixed in position relative 17 to the first mirror. 18 19 Alternatively the second mirror may be movable in 20 position relative to the first mirror. The apparatus 21 may further comprise a guide means for guiding said 22 second mirror in a direction parallel to the first 23 axis, and adjustment means for adjusting the spacing 24 of the first and second mirrors. 25 26 The apparatus may further comprise angular adjustment 27 means adapted to allow angular adjustment of the 28

mirror housing with the X-ray generator tube.

5

1 Preferably the X-ray generator tube is adapted to

- 2 produce an X-ray source at the target having a
- 3 maximum width of less than 50 μm, more preferably
- 4 less than 15 μ m.

5

- 6 According to a second aspect of the present invention
- 7 there is provided a method of delivering X-ray
- 8 radiation to a specimen for the purpose of X-ray
- 9 fluorescence spectrometry using an X-ray generating
- 10 tube, the generating tube having an X-ray exit window
- 11 through which X-ray radiation is emitted,
- 12 the method comprising placing first and second
- 13 paraboloidal X-ray reflecting mirrors between the
- 14 exit window and the specimen,
- using the first mirror to collect diverging X-ray
- 16 radiation at its first end adjacent to the exit
- 17 window and to emit X-ray radiation in a substantially
- 18 parallel beam at its second end,
- 19 and using the second mirror to collect substantially
- 20 parallel X-ray radiation at its first end closest to
- 21 the first mirror and to emit X-ray radiation at its
- 22 second end to a focused spot on the specimen.

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- 24 Preferably the method uses an apparatus according to
- 25 the first aspect of the invention.

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- 27 Embodiments of the invention will now be described,
- 28 by way of example only, with reference to the
- 29 accompanying figures, where:

PCT/GB00/04452 WO 01/38861

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Fig. 1 is a schematic view of two X-ray focusing 1 mirrors used in accordance with the invention to 2 focus an X-ray beam from the source on the X-ray 3 target to the sample to be subject to X-ray 4 fluorescence spectrometry; 5 6 Fig. 2 is a schematic view of an apparatus according 7 to a first aspect of the invention having mirrors 8 fixed relative to each other; and 9 10 Fig. 3 is a schematic view of an apparatus according 11 to a second aspect of the invention having mirrors 12 adjustable relative to each other. 13 14 Referring to Fig. 1 there is shown, in a schematic 15 form and not to scale, an X-ray generating tube 1 16 having an exit window 3, an electron source 6, an 17 anode 7, focusing and stigmator coils 8 and a target 18 2 on which is formed an X-ray source 9. A suitable 19 X-ray generating tube is the MICROSOURCE™ tube 20 described in International Patent Application No 21 PCT/GB97/02022, which is a compact X-ray generator 22 capable of producing small-size, high intensity X-ray 23 sources for low power input. Typically the exit 24 window 3 of the generator 1 is provided in the narrow 25 portion 4 of the X-ray tube about which the X-ray 26 focusing coils 8 are arranged, to the side of the X-27 ray target 2. A first X-ray focusing mirror 11, the 28 collection mirror, is positioned adjacent to the exit 29 window 3 in close coupled arrangement, and a second 30 X-ray focusing mirror 12, the focusing mirror, is 31

arranged coaxially with the first X-ray focusing

- 2 mirror 11, to transfer the . Suitable mirrors 11, 12
- 3 are MICROMIRROR™ X-ray optics as supplied by Bede
- 4 Scientific Instruments Ltd. The mirrors are
- 5 cylindrical specularly reflecting mirrors. Each
- 6 mirror comprises a cylindrical body having an axially
- 7 symmetrical passage extending therethrough. There is
- 8 an aperture at each end of the body which
- 9 communicates with the passage. The reflecting
- surface is on the inside of the long axis of the
- 11 cylinder and has a shape corresponding to a
- paraboloid of revolution about the long axis of the
- 13 cylinder.

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- 15 A paraboloidal profile produces an almost parallel,
- 16 essentially non-divergent beam 14. The interior
- 17 reflecting surface 16 is coated in an exceptionally
- 18 smooth coating of gold or similar in order to provide
- 19 specular reflectivity. Typically the mirror is made
- of nickel and is of the order of 10 to 100 mm in
- 21 length, typically about 30 mm. The outside diameter
- of the mirror is typically 6 mm. The internal
- 23 diameter is typically less than 4 mm. The entry
- 24 aperture is generally smaller than the exit aperture.

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- 26 The two mirrors have an identical profile. The
- 27 source to first mirror distance is in the range 5 to
- 28 50 mm.

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Typically the X-ray generator produces a sub-15 μm 1 spot source on a target of less than 10 mm diameter 2 at a power of up to 30 W. 3 4 The first mirror or paraboloidal optic 11 has a high 5 angle of collection and reflects X-rays into a 6 substantially parallel beam. In practice a beam of 7 divergence less than 40 arc seconds can be achieved. 8 9 The second mirror or paraboloidal optic 12 takes the 10 parallel beam and focuses it down to a spot 15 on the 11 specimen 20 of a size similar to that of the X-ray 12 source, typically a spot with a diameter of less than 13 14 15 µm. 15 The focus 15 of the second optic 12 is typically 16 about 10 to 20 mm away from the far end 13 of optic, 17 giving a much more convenient working distance than 18 is available from prior art XRF apparatus, such as 19 monocapillaries. 20 21 The distance between the two optics 11, 12 may be 22 continuously changed without affecting the focal spot 23 quality, thereby allowing a range of source to sample 24 distances X to be achieved. Typically distance X 25 will be 100 mm or more. 26 27 X-ray optics have very well defined profiles and low 28 surface roughness, and therefore work at very high 29 efficiency. By using paraboloidal mirrors the 30

apparatus of the invention achieves broad band

transmission of X-rays, with an efficiency close to 1 1, since only double reflection of the X-ray 2 radiation is required. 3 4 The invention achieves high X-ray brightness at the 5 focal plane on the target, with a focal spot diameter 6 of as low as 10 μm . 7 8 The apparatus of the invention is truly portable, 9 giving it applications in areas such as forgery 10 detection, which require the apparatus to be taken to 11 12 the specimen. 13 A parabolic surface will produce a parallel beam if 14 the source is placed at the focal point. Conversely 15 a focused beam will be brought to a focus when a 16 parabolic surface is illuminated with a parallel 17 Therefore the method and apparatus of the 18 invention serves to transfer the image of the X-ray 19 spot from the target to the specimen. It should be 20 noted that the target may not be perpendicular to the 21 22 axis of the of the mirrors, so that the effective dimension of the image on the target, when viewed 23 along the axis of the mirrors, is less than the 24 25 actual dimension on the target. 26 27 The focal spot size at the specimen is thus primarily determined by the spot size on the target of the X-28

ray tube. Since the first mirror produces a parallel

beam, the focal spot size at the specimen is, within

practical limits, independent of the distance of the

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second mirror along the beam axis. Therefore the

- 2 second mirror can be placed at the required distance
- 3 from the first in order to suit the geometrical
- 4 requirements of the equipment.

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- 6 Figs. 2 and 3 show two schematic arrangements for
- 7 housing the apparatus of the invention.

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- 9 In the simplest case, shown in Fig. 2, the collector
- and focusing mirrors 11, 12 are aligned with each
- other and are fixed within a cylindrical housing 30.
- 12 The housing is aligned relative to the X-ray source
- 9, shown purely schematically in Figs. 2 and 3, on
- 14 the beam axis 32, either fixedly or adjustably. The
- housing 30 may be subject to a partial or total
- vacuum, to improve the efficiency of the mirrors and
- 17 reduce energy absorption as the X-rays pass through
- the gas in the housing 30. It is to be understood
- 19 that in practice the source 9 is part of an X-ray
- generating tube 1 (not shown in Figs. 2 and 3).

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- 22 In use the housing 30 is placed adjacent to the X-ray
- 23 source, and a control mechanism 35 allows fine
- 24 adjustment of the position of the housing 30 in the
- \mathbf{x} , \mathbf{y} and \mathbf{z} directions so that the axis 32 of the
- 26 mirrors is accurately aligned with the X-ray source 9
- 27 and directed to the specimen 20. The control
- mechanism 35 may comprise any suitable mechanisms
- 29 which permit fine translational adjustment, such as
- 30 lead screws or Vernier controls.

In the example of Fig. 3, each mirror 11, 12 is 1 provided with a separate housing 40, 41. 2 housings 40, 41 may further be contained in an outer 3 housing, not shown, which may be partially or 4 completely evacuated. The apparatus allows alignment 5 of the second mirror 12 relative to the first mirror 6 11 and translation of the second mirror 12 along the 7 beam axis 43 by means of control mechanism 44. 8 9 Alignment of the whole mirror assembly relative to 10 the X-ray source 9 is possible by means of control 11 mechanism 45. Mechanisms 44 and 45 are similar to 12 mechanism 35 described with reference to Fig. 2, and 13 are not described further. 14 15 Although the invention has been described with 16 reference to a microfocus X-ray generator, the 17 invention can be used with any suitable X-ray 18 generator which is capable of producing a small 19 source of sufficient intensity. 20 21 The mirror housing 30, 40 may be attached to the X-22 ray tube 1 or may be positioned independently. 23 24 These and other modifications and improvements can be 25

incorporated without departing from the scope of the

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invention.

PCT/GB00/04452 WO 01/38861 12

1 CLAIMS

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- An apparatus for carrying out X-ray fluorescence 3
- spectrometry comprising an X-ray generating tube (1) and 4
- two paraboloidal X-ray reflecting mirrors (11, 12), the 5
- generating tube having an X-ray source (9) and an X-ray 6
- exit window (3) through which X-ray radiation from said 7
- source is emitted, 8
- the first mirror (11) being aligned on a first axis (32, 9
- 43) and positioned in close coupled arrangement adjacent 10
- to the exit window (3), the second mirror (12) being 11
- aligned on said first axis and being positioned in spaced 12
- apart relationship to the first mirror (11), 13
- the first mirror (11) being adapted to collect diverging 14
- X-ray radiation at its first end adjacent to the 15
- collecting window (3) and to emit X-ray radiation in a 16
- substantially parallel beam at its second end, 17
- the second mirror (12) being adapted to collect 18
- substantially parallel X-ray radiation at its first end 19
- closest to the first mirror and to emit X-ray radiation 20
- in a focused beam at its second end. 21

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- An apparatus according to Claim 1, wherein the first 23 2.
- and second mirrors (11, 12) are cylindrical specularly 24
- reflecting mirrors. 25

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- An apparatus according to Claim 1 or 2, wherein the 27
- first end of the first mirror (11) is positioned between 28
- 5 and 50 mm from the X-ray source (9). 29

- An apparatus according to any preceding Claim, 31 4.
- wherein the apparatus further comprises a housing 32
- containing the first and second mirrors. 33

2 5. An apparatus according to any preceding Claim,

3 wherein the second mirror is fixed in position relative

4 to the first mirror.

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6 6. An apparatus according to any of Claims 1 to 4,

7 wherein the second mirror is movable in position relative

8 to the first mirror.

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10 7. An apparatus according to Claim 6, further

11 comprising a guide means for guiding said second mirror

in a direction parallel to the first axis, and adjustment

means for adjusting the spacing of the first and second

14 mirrors.

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16 8. An apparatus according to any preceding Claim,

17 further comprising angular adjustment means adapted to

18 allow angular adjustment of the mirror housing with the

19 X-ray generator tube.

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21 9. An apparatus according to any preceding Claim,

wherein the X-ray generator tube is adapted to produce an

23 X-ray source at the target having a maximum width of less

24 than 50 μ m, more preferably less than 15 μ m.

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26 10. An apparatus according to any preceding Claim,

27 wherein the apparatus is portable and the X-ray generator

28 is a microfocus generator.

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30 11. A method of delivering X-ray radiation to a specimen

for the purpose of X-ray fluorescence spectrometry using

32 an X-ray generating tube, the generating tube having an

1 X-ray exit window through which X-ray radiation is

- 2 emitted,
- 3 the method comprising placing first and second
- 4 paraboloidal X-ray reflecting mirrors between the exit
- 5 window and the specimen,
- 6 using the first mirror to collect diverging X-ray
- 7 radiation at its first end adjacent to the exit window
- 8 and to emit X-ray radiation in a substantially parallel
- 9 beam at its second end,
- 10 and using the second mirror to collect substantially
- parallel X-ray radiation at its first end closest to the
- 12 first mirror and to emit X-ray radiation at its second
- 13 end to a focused spot on the specimen.

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- 15 12. A method according to Claim 11, wherein the first
- and second mirrors (11, 12) are cylindrical specularly
- 17 reflecting mirrors.

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- 19 13. A method according to Claim 11 or 12, wherein the
- 20 first end of the first mirror (11) is positioned between
- 5 and 50 mm from the X-ray source (9).

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- 23 14. A method according to any one of Claims 11 to 13,
- 24 wherein the focused spot on the specimen has a maximum
- 25 dimension of 50 μ m.

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- 27 15. A method according to any one of Claims 11 to 14,
- 28 further comprising the step of adjusting the spacing of
- 29 the first and second mirrors to produce a focused spot on
- 30 the specimen.

- 32 16. A method according to any one of Claims 11 to 15,
- 33 wherein the X-ray generator is a microfocus generator.

